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QUARTERLY REPORT - JUNE-AUGUST 1962

CONTRACT NUMBER AF 04 (611) - 8383
PROGRAM STRUCTURE NUMBER 6399 - 623A
PROJECT NUMBER 623A

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National Carbon Co.

October 11, 1962

6593d Test Group
(Development)
Edwards Air Force Base, Calif.
For: DGSMA

Subject: Contract AF 04 (611) - 8383
Quarterly Report

Gentlemen:

Enclosed are the required copies of the subject Report for the period ending August 31, 1962. The attached distribution list indicates copies for other recipients as indicated in the contract.

Very truly yours,

D. E. Muir
Assistant Secretary

DEM:jem
Encs.

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4 June 1962 - 31 August 1962
Contract No. AF 04 (611) - 8383
Program Structure No. 6399 - 623A
Project Number 623A

"FABRICATION & TESTING OF 103" O.D. X 34" I.D. X 70" LONG GRADE CFW GRAPHITE"

QUARTERLY REPORT

QUARTERLY REPORT - JUNE-AUGUST 1962

CONTRACT NUMBER AF 04 (611) - 8383
PROGRAM STRUCTURE NUMBER 6399 - 623A
PROJECT NUMBER 623A

INTRODUCTION

In support of the large Solid Rocket Booster Program, a need exists for graphite nozzle throat inserts in sizes larger than those previously produced by the graphite industry. As a result of this need, National Carbon Company was awarded Contract No. AF 04 (611) - 8383 to fabricate and test three 103" O.D. x 34" I.D. x 70" long Grade CFW graphite cylinders. One objective of this contract is to demonstrate the feasibility of fabricating monolithic graphite for rocket nozzle throat inserts in sizes 100" diameter and larger. A further objective calls for a physical property testing program, both at ambient and elevated temperatures, to assist in more fully characterizing such materials.

The program to be conducted under this contract encompasses three complementary phases. They are (1) make the necessary facility modifications or additions required for producing graphite in sizes larger than any previously manufactured, (2) fabricate the required graphite cylinders, and (3) conduct the required testing to enable characterization of this large graphite size. Also, under Phase 3, attempts will be made to investigate the feasibility of using radiographic and ultrasonic techniques to non-destructively examine structural integrity of large graphite sizes.

Production of the graphite required under this contract will be carried out primarily in new manufacturing facilities financed and installed by National Carbon Company. Normal physical property testing will be conducted at National Carbon Company Laboratories, with elevated temperature measurements being made at the Advanced Materials Laboratory, Lawrenceburg, Tennessee, operating under Contract AF 33 (616) - 6915.

This is the first Quarterly Contract Report and covers the period from contract approval (June 4, 1962) through August 31, 1962.

SUMMARY

To enable production of the larger graphite sizes indicated as required for aerospace applications, National Carbon Company has financed and installed new processing facilities including a 14,000 ton press. Construction and installation of this equipment was started during 1961 and has been essentially completed in the past three months.

Because of the graphite size and nature of equipment involved, considerable development work has been carried out in the early stages of this contract to enable a "scale up" in size using existing graphite technology.

- more -

Green carbon cylinders approximately 105" O.D. x 32" I.D. x 83" long were successfully molded ~~in July~~ using ~~the~~ standard base materials, ~~specified for Grade GFW.~~

The molded cylinders were loaded into baking furnaces ~~during early August,~~ and the approximately twelve-week long heating-cooling cycle was initiated. The baking operation is progressing in accord with the pre-determined program and will terminate in October.

A review of the progress to date in facility construction, technological scale up, and material processing ~~indicates completion of the various phases of this contract are on schedule~~ is presented.

DISCUSSION

New Facilities and Equipment

Construction and installation of the largest graphite processing equipment made to date have been completed in the last three months. To facilitate the production of the three graphite cylinders required under this contract, as well as materials for future aerospace requirements, this equipment has been installed at the National Carbon Company Republic Plant, Niagara Falls, New York. An 81 foot high main building has been erected to house these facilities. The major items include a 14,000 ton molding press to form pieces up to 200 inches in diameter, gas baking and graphitizing furnaces, and impregnating autoclaves, as well as a special overhead crane, and handling equipment for graphite weighing as much as thirty tons.

A 105" diameter steel mold, jacketed for temperature control of the green carbon mix during the molding operation, was designed and fabricated. In conjunction, a 32" diameter steel mandrel was also manufactured. This equipment was produced under the contract. Trial pieces were molded prior to the first production run to check out equipment operation, temperature controls, and processing details. As a result of this work, the molding equipment performed satisfactorily during forming of four full-sized cylinders. One of these molded cylinders is shown in Figure 2.

Installation of the recirculating, natural gas-fired furnace being used to bake these large cylinders was completed in July. This furnace was designed with automatic controls and thermocouple coverage. During the baking operation, it is imperative that very low temperature gradients be maintained within the pieces. Operation and control of this furnace was thoroughly checked out prior to loading the molded stock. The furnace heating cycle was started in early August. Figures 2 and 3 show this baking furnace before and after loading the 105" diameter cylinders.

The equipment necessary for impregnating this stock to increase density and strength was completed in June. This includes the pressure vessel autoclave, pre-heater, impregnant storage facilities, and vacuum equipment. Several trial production runs have been made with this equipment, using standard stock sizes to acquire the necessary experience for treating the larger 103" diameter stock. Based on the information obtained during these break-in runs, it is anticipated that thorough impregnation of the 103" diameter CFW will be achieved.

Likewise, the facilities for heating the stock to graphitizing temperatures (2600-3000°C) have been designed and installed. An initial trial run has been made and all components are functioning properly.

Processing

National Carbon Company has previously developed Grade CFW as a high density, impregnated graphite from which the large size components required in the aerospace industry can be fabricated. Several large booster motor test firings have been successfully made using nozzle throats fabricated from 48" diameter Grade CFW graphite. Production of the 103" O.D. x 34" I.D. x 70" long Grade CFW requires a major scale up of standard graphite production processes and developed technology in the manufacture of the 48" diameter material.

After receipt of the supporting equipment necessary for forming the 103" diameter cylinders, several trial pieces were molded to optimize forming techniques, raw material mixing and handling operations, and to develop skills in handling this larger size. When molding the green mix materials, it is important that proper uniform temperatures be maintained throughout the mold, thus keeping the carbonaceous binder at the proper plastic level for good bonding. Controlled cooling is likewise important to prevent distortion and cracking.

When forming techniques were finalized, the dry aggregate and carbonaceous binder materials for Grade CFW were prepared and mixed in accord with standard manufacturing specifications. The initial four full-sized pieces were then formed without incident. The property data for these "green" molded pieces is shown in Table I.

Stock identification		7L2	8L2	9L2	10L2
Date molded		7/17/62	7/19/62	7/20/62	7/27/62
Average outside diameter	(in.)	105.25	105.19	105.19	105.21
Average inside diameter	(in.)	32.58	32.63	32.63	32.67
Average length	(in.)	84.36	85.00	83.41	85.38
Weight	(lbs.)	40,980	40,300	39,970	40,710
Bulk Density	(g/cc)	1.71	1.71	1.69	1.68

[illegible] densities shown above are considered highly satisfactory for the "green" molded stage. The higher density of Grade AXF graphite is obtained through impregnation at subsequent processing stages.

These four molded pieces were loaded in the gas-fired baking furnace (Figure 3) the first week of August. Furnace pack material consisting of pre-conditioned calcined petroleum coke and sand was placed in the void space around the molded stock. This pack is designed to prevent distortion of the molded stock while going through a plastic range in the early stages of baking. The baking operation is the first elevated temperature process in the manufacture of graphite. It is during this cycle, while heating to approximately 750°C, that the volatiles are driven out of the carbonaceous binder material and an infusible carbon bond is formed. The temperature changes throughout this operation must be closely controlled and carried out very slowly. This permits safe evolution of gasses, without cracking the stock, and allows the subsequent material contraction. The heating and cooling cycle which constitutes the baking operation must be carried out with changes of only a few degress per day and requires approximately twelve weeks total time. The baking cycle for this contract material was started on August 7, 1962, based on a pre-determined temperature program, and is progressing exceedingly well. It is expected to be terminated about mid-October.

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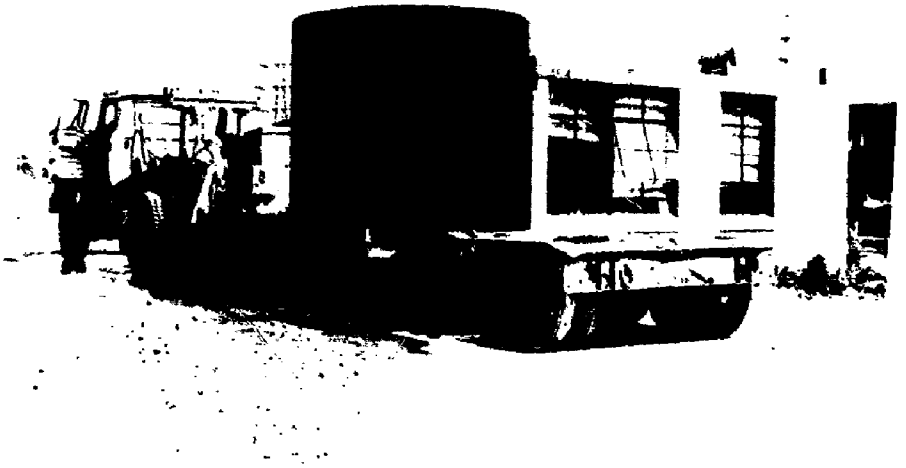

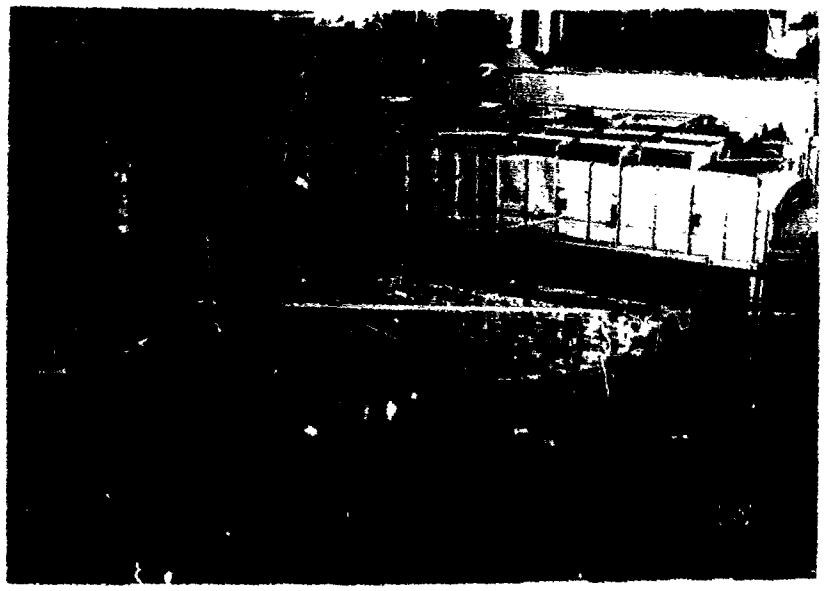

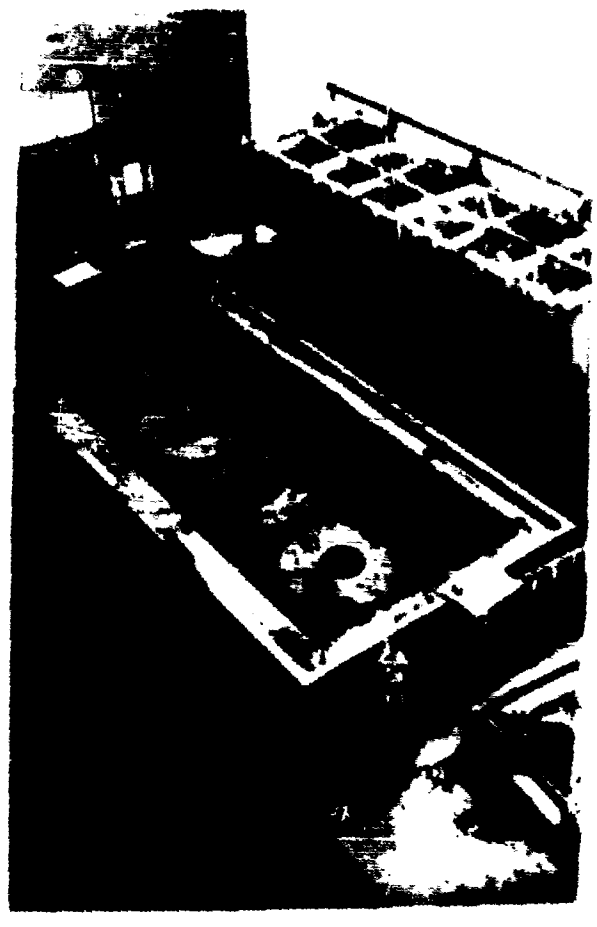

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